# EFFECT OF DRYING AND REWETTING CYCLES OF CELLULOSIC FIBRES ON RESISTANCE OF CEMENTITIUS COMPOSITES


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## INTRODUCTION:

It is well known that drying and rewetting cycles principally cause shrinkage of the natural fibres due to the formation of hydrogen bonds in cellulose [1]. This irreversible effect is known as "hornification" and is quantified as the percentage reduction in water retention values (WRV). The reduction in the WRV of the hornificated fibres could have beneficial effects on vegetable fibre reinforced cement composites. On one hand, the hornificated fibres will have higher dimensional stability, and thus higher fibre-matrix adherence is expected. On the other hand, as a consequence of the lower WRV of these hornificated fibres, a reduction in the formation of incrustations of calcium hydroxide on the surface and lumen of the fibres and consequently a reduction in the degradation of the cellulose in the cementitious matrix are expected.

In this study two types of cellulosic fibres -chemical pulp from softwood and cotton linters- previously subjected to hornification process have been used to prepare cement mortar composites. The resistance of these composites was tested after both 28 days of cure treatment and four wet-dry cycles. Results indicated that the previous treatment of fibres (hornification process) has beneficial effects on the resistance of the resulting cementitious composites.

## MATERIALS AND EXPERIMENTAL PROCEDURES:

**MATERIALS:**

- *CEMENT:* Type I Portland cement according to UNE-EN 197-1:2000
- *CELLULOSIC FIBRES:*
  - ⇒Unbleached softwood kraft pulp (Pinus insignis) was supplied by Smurfit Kappa Nervión, S.A. (Spain). In order to obtain pulps with different percentages of lignin, the unbleached pulp was subjected to an Elemental Chlorine Free (ECF) bleaching sequence. The reference of the samples obtained was: unbleached pulp **[U]**, oxygen delignificated pulp **[O]**, semi-bleached pulp **[OD]**, and fully bleached pulp **[OD(EP)D]**.
  - ⇒Cotton linters were supplied by Celsur (Cotton South, S.L., Spain).

**HORNIFICATION PROCESS PERFORMED ON THE FIBRES:**

The fibres were subjected to four drying and rewetting cycles and the resulting pulps characterized. The sequence was as follows:
(1) Drying in an oven with air recirculation at 60 °C for 7 h;
(2) Rewetting by soaking overnight
(3) Disintegration of the wet pulp for 30,000 revolutions
(4) Filtration of the pulp suspension.

**COMPOSITE PREPARATION:**

Cement composites containing 4 wt. % of fibres were prepared with both the hornificated and unhornificated ones.
The composites were moulded in the size required for testing (40x20x160 mm) and compressed at 0,4 MPa during 24 h. All the specimens prepared were cured during 28 days (RH>95%, ambient pressure, 20ºC). Half of the specimens to test were aged with a process consisting in four wet-dry cycles.

**CHARACTERIZATION OF THE FIBRES:**

- **Water retention value** (WRV) according to ISO 23714:2007
- **Morphology** using a Jeol-820 Scanning Electron Microscope.
- **X-Ray diffraction (XRD)** performed on a Siemens D-500 diffractometer.
- **Thermogravimetric analysis (TGA)** performed on a Mettler Toledo TGA/SDTA 851[e].

**CHARACTERIZATION OF THE COMPOSITES:**

The resistance of the composites was evaluated with flexural tests.

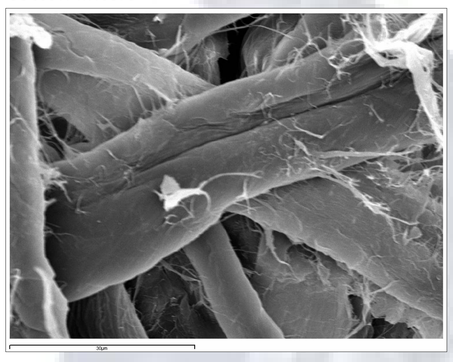
## RESULTS:

### CHARACTERIZATION OF THE FIBRES

*Values of WRV after the drying and rewetting cycles*

| *Values in %* | *Wet/dry cycles* | | | | | Hornification (%) |
|---|---|---|---|---|---|---|
| | *Initial* | *I* | *II* | *III* | IV | |
| **Unbleached** | 126 | 112 | 102 | 97 | 90 | 28,6 |
| **Oxygen Delignificated** | 122 | 107 | 95 | 91 | 86 | 29,5 |
| **Semibleached** | 121 | 94 | 89 | 82 | 79 | 34,7 |
| **Fully bleached** | 126 | 95 | 84 | 78 | 75 | 40,5 |
| **Cotton linters** | - | 58 | 54 | 52 | 50 | 13,8 |

***SEM*** *micrographs of the unhornificated cotton linters*

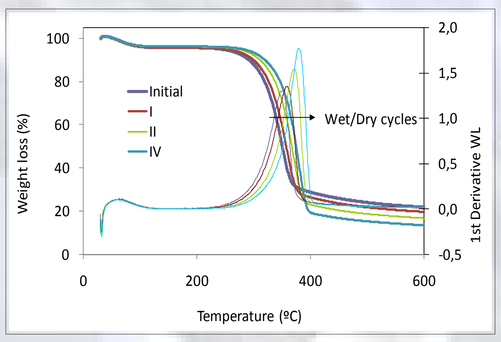


***SEM*** *micrographs of the hornificated cotton linters*

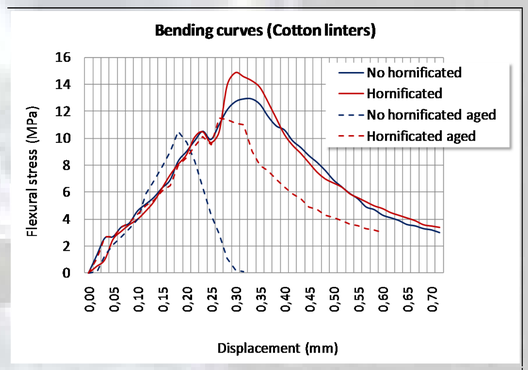
*Crystalinity index with the drying and rewetting cycles obtained from X-ray diffractograms XRD patterns of the softwood pulps and cotton linters*

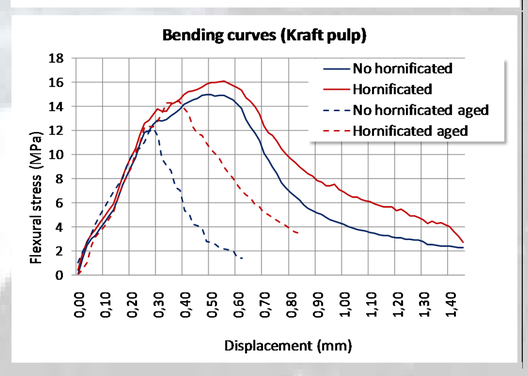
| | Crystallinity index (%) | | | | |
|---|---|---|---|---|---|
| *Wet/dry cycles* | *Initial* | *I* | *II* | *III* | IV |
| **Unbleached** | 56.8 | 57.1 | 57.8 | 60.4 | 58.0 |
| **Oxygen Delignificated** | 67.4 | 68.8 | 68.1 | 68.2 | 67.1 |
| **Semibleached** | 66.7 | 67.1 | 68.4 | 69.2 | 72.7 |
| **Fully bleached** | 68.2 | 67.5 | 69.3 | 67.9 | 67.2 |
| **Cotton Linters** | 78.5 | 78.7 | 82.0 | 81.0 | 80.9 |

*TGA thermograms of the unbleached softwood with the drying and rewetting cycles.*




### CHARACTERIZATION OF THE COMPOSITES




## CONCLUSIONS:

- WRV and curl index values decreased with the drying and rewetting cycles, with this effect being more significant in the softwood bleached pulps. Cotton linters showed the lowest value for the percentage of hornification. This decrease in the WRV and the shrinkage of the fibres will improve their dimensional stability against environmental changes in cement mortar composites, reduce the water gradient in the fibre–matrix interface, and will probably decrease the crystallization of calcium hydroxide particles on the surface of the fibres.
- The hornificated fibres exhibited enhanced thermal properties, increasing the thermal degradation temperature with the number of drying and rewetting cycles.
- The flexural resistance increased in the composites prepared with the hornificated fibres. This behaviour was observed on both the unaged and aged composites.


## ACKNOWLEDGMENTS:

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## REFERENCES:


[1] Weise, U., & Paulapuro, H. (1999). Effect of drying and rewetting cycles on fibre swelling. Journal of Pulp and Paper Science, 25(5), 163–166.